AMENDMENT NO. 1 MARCH 2021

TO

IS 2833 : 2019 ANILINE, TECHNICAL — SPECIFICATION

(Second Revision)

(*Page* 1, *Clause* **4.1**) — Substitute the following for the existing clause:

‘4.1 Packing

The material shall be packed in suitable drums conforming to IS 1783 (Part 1), road tankers, ISO containers or suitable drums/containers certified by accredited agency.’

(*Page* 1, *Clause* **4.2**) — Substitute the following for the existing clause:

‘4.2 Marking

Each container shall be securely closed and shall bear the following information:

a) Name of the material;
b) Name of the manufacturer;
c) Lot or batch number, when applicable;
d) Tare, net and-gross mass; and
e) POISON [red label, printed on white background conforming to IS 1260 (Part 1)]

(*Page* 4, Annex **B**) — Substitute the following for the existing Annex:

‘ANNEX B

(Table 1)

DETERMINATION OF ANILINE AND NITROBENZENE CONTENT

B-0 GENERAL

To determine purity of aniline as well as impurity of nitrobenzene in single gas chromatography run, test method given at **B-1** may be followed and to determine purity of aniline as well as impurity of nitrobenzene, separately, test methods given at **B-2** and **B-3** may be followed.

B-1 PRINCIPLE OF GAS CHROMATOGRAPHY

Gas chromatography is a separation technique based on the difference in the distribution of components in a mixture between a mobile gas phase and a stationary phase. As it passes through the stationary phase, the mobile phase elutes the separated components one by one. The eluted components in the mobile phase are detected by a detector block and recorded by a [mv] recorder in terms of the variables of the components.

B-1.1 GC Equipment

Gas chromatography system with flame ionization detector and auto injector.

B-1.2 GC Parameters

Column:	Stationary phase (capillary column), having 86 percent dimmethyl polysiloxane and 14 percent cyanopropyl phenyl as packing material (for example DB-1701 and its equivalent) with 30 m Length x 0.25 mm ID x 1.0 µm film thickness	
Injector:	Split	
	Temperature:	275 °C
	Split ratio:	30
Carrier gas:	Nitrogen or helium with flow control mode pressure	

Carrier gas flow rate: 30 ml/min

Carrier gas pressure: 100 kpa

Column oven temperature 100°C

Oven temperature:

Heating Rate (°C/min)	*Temperature (°C)*	*Hold Time (min)*
–	100	2
10	230	15

Run time: 30 min

FID Parameters

Hydrogen flow: 40 ml/min

Air flow: 400 ml/min

Temperature: 275 °C

NOTE — Aging of the column can influence the separation so that the conditions parameters like temperature and flow may have to be changed.

B-1.3 Standard and Sample Preparation

Use neat standard and sample for injection.

B-1.4 Injection Volume

Aniline standard and sample: 1.0 µl

Nitrobenzene: 1.0 µl

B-1.5 Retention Time

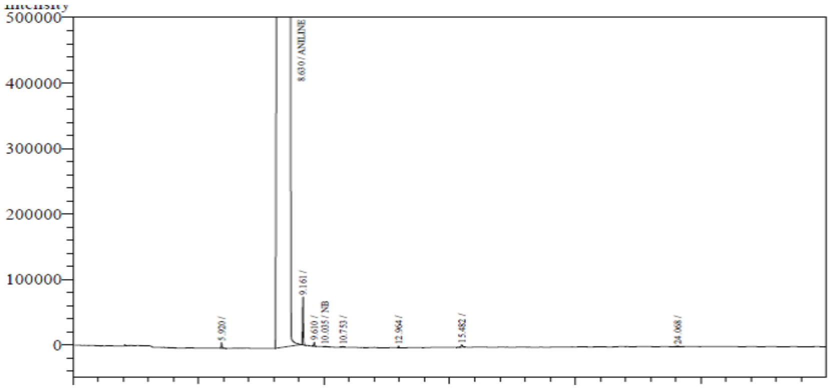
Aniline: 8.63 min

Nitrobenzene: 10.03 min

B-1.5 Calculation

Take value of aniline and nitrobenzene by area normalization.

Chromatogram



B-2 DETERMINATION OF ASSAY OF ANILINE

This method is used for the determination of purity of aniline. A sample of aniline is injected on a gas chromatographic capillary column. After separation of the necessary components, the results are calculated using the internal standard method.

B-2.1 Apparatus

B-2.1.1 *Gas Chromatograph,* equipped with an FID detector, a split-splitless injector and a suitable electronic integrator/software.

B-2.1.2 *Capillary Column*, fused silica, with 100 percent polyethylene glycol (PEG) base deactivated.

B-2.1.3 *Column Size,* length: 30 m, I.D.: 0.32 mm, film thickness: 0.32µm.

B-2.1.4 *Syringe for Gas Chromatography*, volume 5 or 10 µl.

B-2.1.5 *Analytical Balance*, accurate to 0.1 mg.

B-2.2 Reagent — Use AR grade chemicals

a) Aniline
b) Cyclohexanol

c) Cyclohexanone
d) Cyclohexylamine
e) Cyclohexylaniline
f) m-Toluidine
g) p-Toluidine
h) p-Phenol
j) m-Phenylenediamine
k) Diphenylamine
m) o-Dichlorobenzene (ODCB), (internal standard)

B-2.3 Sampling

The sample must be taken in a clean, dry, 500 ml glass bottle. Do not expose the sample to air.

SAFETY

Aniline has high acute toxicity. Toxic levels may be reached by adsorption of the liquid through skin contact and/or inhalation of vapors. The threshold limit value (TLV) for aniline is 2 ppm. Work under a fume hood so as to avoid inhaling aniline vapors and use adequate gloves wherever possible to avoid contact with skin.

B-2.4 GC Parameters

Carrier gas : Hydrogen
Pressure at head of column : 15 psi
Fuel gas and flow rate : Hydrogen: 40 ml/min, Air: 400 ml/min
Make up flow : As per the requirement of instrument make/ model.
Sample preparation : Direct (as such sample)
Injection volume : 1µl
Injector : Split injector
Split flow : 100 ml/min
Detector : FID

Temperature programme of oven:

Injector Temperature (°C)	*Detector Temperature (°C)*	*Oven*		
		Temperature (°C)	*Hold Time (min)*	*Ramp Rate (°C/min)*
250	250	110	4	15
		200	12	0

B-2.5 Calibration

Prepare a standard solution containing the components given below, in the expected concentration range. A typical example of standard solution is given below for reference.

Component	*Mass, g*	*Concentration, percent (w/w)*
Benzene	0.0250	0.0249
Cyclohexylamine	0.0250	0.0249
Cyclohexanol	0.0250	0.0249
Cyclohexanone	0.0500	0.0499
Aniline	100.0000	99.7258
p-Toluidine	0.0250	0.0249
m-Toluidine	0.0250	0.0249
Phenol	0.0250	0.0249
Cyclohexylaniline	0.0250	0.0249
m-phenylinediamine	0.0250	0.0249
Diphenylamine	0.0250	0.0249

B-2.5.1 Make sure the GC instrument is adjusted to the conditions stated as above. Place 10 ml aniline standard sample in 10 ml volumetric flask. Add 2 micro Lit. of the internal standard (ODCB) using 10 micro Lit syringe. Shake the sample to mix. Inject 1.0 µl of the standard at least three times and obtain the chromatograms and integrate the area under each peak excluding aniline. Calculate the response factor of each impurity as under:

$$\text{Response Factor (RF)} = \frac{(A_1 \times C_1)}{(A_2 \times C_2)}$$

where

A_1= Peak area of internal standard

A_2= Peak area of impurity

C_1= Concentration of impurity

C_2= Concentration of internal standard (use density 1.018 gm/ml of calibration blend and density 1.300 gm/ml for internal standard (ODCB).

B-2.5.1.1 Find the arithmetic mean of the factors calculated for each component. Each factor is calculated to 4 decimal places. When using the factors, the calculation of the concentration of the sample is done using the mean obtained. Suitable software for auto calibration with respect to mass of calibration standard may also be used.

B-2.6 Procedure of Analysis

B-2.6.1 Make sure the GC instrument is adjusted to the conditions stated as above. Place 10 ml aniline sample in 10 ml volumetric flask. Add 2 micro Lit. of the internal standard (ODCB) using 10 micro Lit syringe. Shake the sample to mix. Inject 1.0 µl of the sample and obtain the chromatograms and integrate the area under each peak excluding aniline. Calculate the concentration of impurity as under:

$$\text{Concentration of Impurity} = \frac{A_2 \times RF \times C_2}{A_1}$$

where

A_1= Peak area of internal standard

A_2= Peak area of impurity

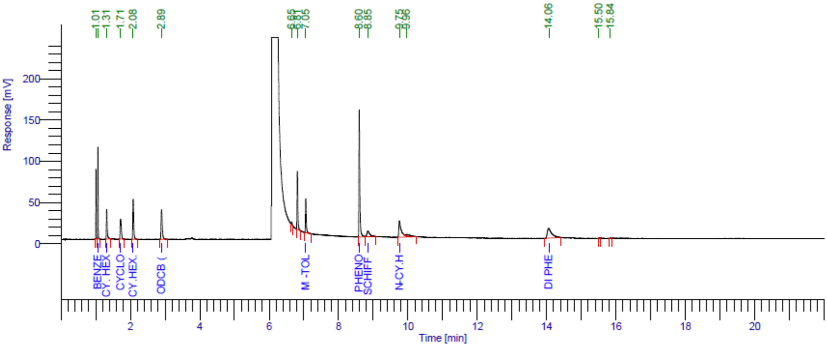
C_1= Concentration of impurity

C_2= Concentration of internal standard

Suitable software for auto calculation with respect to calibration standard may also be used.

B-2.6.2 *Calculation*

Assay percent, Aniline = 100 – Sum of concentration of all impurity.



Sl No.	*Component Name*	*Retention Time*
1	Benzene	1.04
2	Cyclohexylamine	1.28
3	Cyclohexanol	1.65
4	Cyclohexanone	1.97
5	o-dichlorobenzene (ODCB) – internal standard	2.75
6	Aniline	5.83
7	p-Toluidine	6.45
8	m-Toluidine	6.62
9	Phenol	8.43
10	n-Cyclohexylaniline	9.63
11	m-phenylinediamine	12.80
12	Diphenylamine	13.90

B-3 DETERMINATION OF NITROBENZENE CONTENT IN ANILINE

This method is used for the determination of nitrobenzene at ppm levels in aniline. A sample of aniline is injected in a gas chromatographic column. After separation of the necessary components, the results are calculated using the internal standard method.

B-3.1 Apparatus

B-3.1.1 *Gas Chromatograph*, equipped with an ECD detector, a packed column injector and a suitable electronic integrator/software.

B-3.1.2 *Packed Column*, XE -60 on Chromosorb PAW, mesh size: 60/80, [XE -60: 25 percent 2-cyanoethyl- 75 percent methyl(poly)siloxane]

B-3.1.3 *Column Size,* Length: 3 m, O.D.: 1/8”

B-3.1.4 *Syringe for Gas Chromatography*, volume 5 or 10 µl.

B-3.1.5 *Analytical Balance*, accurate to 0.1 mg.

B-3.2 Reagent — Use AR grade chemicals

a) Aniline
b) Nitrobenzene

B-3.3 Sampling

The sample must be taken in a clean, dry, 500 ml glass bottle. Do not expose the sample to air.

SAFETY

Aniline has high acute toxicity. Toxic levels may be reached by adsorption of the liquid through skin contact and/or inhalation of vapors. The threshold limit value (TLV) for aniline is 2 ppm. Work under a fume hood so as to avoid inhaling aniline vapors and use adequate gloves wherever possible to avoid contact with skin.

B-3.4 GC Parameters

Carrier gas : Nitrogen
Makeup gas : Nitrogen
Column flow : 30 ml /min.
Make flow : 35 ml /min.
Sample preparation : Direct (as such sample)
Injection volume : 1µl
Detector : ECD (Electron Capture Detector)
Detector temperature : 350°C
Injector : Packed injector,
Injector temperature : 240°C
Oven temperature : 170°C

B-3.5 Calibration

Prepare a standard solution containing the component given below, in the expected concentration range. A typical example of standard solution is given below for reference.

Component	*Mass, g*	*Concentration, percent (w/w)*
Aniline	100.00	–
Nitrobenzene	0.0010	0.0010 (10 ppm)

B-3.5.1 Make sure the GC instrument is adjusted to the conditions stated as above. Inject 1.0 µl of the standard at least three times and obtain the chromatograms and integrate the area of nitrobenzene peak. Calculate the response factor of each impurity as under:

Response Factor (RF) = A_1/C_1

where

A_1= Peak area of nitrobenzene in calibration standard
C_1= Concentration of nitrobenzene in calibration standard

B-3.6 Procedure of Analysis

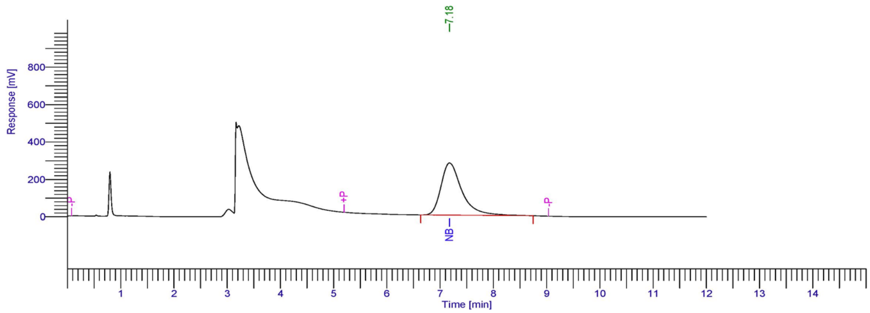
B-3.6.1 Make sure the GC instrument is adjusted to the conditions stated as above. Inject 1.0 µl of the sample and obtain the chromatograms and integrate the area of nitrobenzene peak. Calculate the concentration of impurity as under:

Nitrobenzene content in ppm = A_2/RF

where

A_2 = Peak area of nitrobenzene of sample.

Suitable software for auto calculation with respect to calibration standard may also be used.



Retention time of nitrobenzene: 7.16 min.

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc), calibration technique (internal standard, external standard, area normalization, percent area etc), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.’

(PCD 26)

Publication Unit, BIS, New Delhi, India